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# DEVELOPMENT OF AN ULTRSONIC TNT SLURRY ANALYZER

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14. ABSTRACT

The goal of this work was to develop, install, and test an ultrasonic instrument for continuous, nondestructive evaluation of TNT mixtures inside a production melt kettle. A first instrument was installed and tested at a pilot-scale melt kettle in building 810 at the U.S. Army Armament Research, Development and Engineering Center, Picatinny Arsenal, NJ. This instrument was then lengthened for installation in a full-sized melt kettle on Line 3a at the Iowa Army Ammunition Plant, Middletown, Iowa (operated by American Ordnance). The instrument includes an ultrasonic transmitter and receiver encased in a stainless steel probe, and the electronics to interpret the measured ultrasonic signals. The instrument provides continuous information on the amount of solid TNT in the solid/liquid (molten) TNT mixture in the kettle. This information will be used to improve the quality, safety, and costs of manufacturing TNT munitions.

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## INTRODUCTION

Many of the Army's warheads are manufactured as castings from melted energetic materials. Changes in the solids/liquid mix can lead to inconsistencies in the final cast product after it cools. Inconsistencies can lead to cracking and other defects that affect detonation.

The current process for the manufacture of TNT loaded munitions involves the preparation of TNT slurry (solid and molten TNT mixture) in a large, 350-gal melt kettle. About 25 to 30% solids by weight are slowly added to molten TNT already in the kettle; i.e., the "heel." After a fixed heating time, the kettle valve is opened and projectiles are batch loaded. Prior to this work, no instrument existed to measure the actual solids in the TNT slurry mixture. The only method available was to predetermine the amount of solids to add to the heel. In order to maintain a slurry mixture, the kettle must be heated to a temperature above the solids TNT melting point. This results in constant melting of TNT solids (burning off) in the melt kettle, and thus the percent of TNT solids is continually reduced. Maintaining a proper solid-liquid mixture is thus operator dependent and can vary significantly for different batches. This can affect the quality of the casting and result in defects such as piping, cracking, and base separation, which increase the cost of production. In addition, firing a defective munition endangers the soldier in the field.

University of Denver Research Institute (DRI) scientists have developed instruments that measure the amount of solids in a liquid using ultrasound waves. These waves are high frequency pressure waves that travel though materials and reflect from boundaries. Ultrasound is a safe means for measuring energetic materials because of the low energy in the waves. For additional safety, the ultrasonic sensors can be isolated from the process mixture behind walls of stainless steel.

The solids measurement technique relies on the different ultrasonic properties of the TNT in solid and liquid form. The amount of solid material in the mix effects the transmission of the ultrasonic waves through a small volume of the mixture. By measuring these transmission properties, the amount of solids can be determined.

This ultrasonic technique was incorporated into a new instrument that measures the solids in the TNT slurries while they are in the mixing kettle. Ultrasonic transducers are encased inside a probe and do not contact the TNT mixture. The probe is inserted into the process kettle through an opening at the top. An electronic system located in the control room is used to process the ultrasonic signals and calculate solids composition of the slurry. The probe and the associated electronics system are termed the "TNT Slurry Analyzer," or TSA for short. This document details the installation and testing of the TSA at the Iowa Army Ammunition Plant (IAAP), Middletown, Iowa. Details of the earlier testing of the first analyzer at the U.S. Army Armament Research, Development and Engineering Center (ARDEC), Picatinny Arsenal, New Jersey can be found in the TSA Description and Operating Manual (ref 1).

## MEASUREMENT PRINCIPLES

The TSA determines solids composition by measuring ultrasonic time-of-flight (TOF) and amplitudes of signals passing through a gap that is filled with the process fluid (ref. 2). Figure 1 shows a photograph of the gap on the TNT slurry probe. The TOF is the time the ultrasonic signal takes to travel from one sensor, across the fluid in the gap, to the receiving sensor. This TOF is dependent on the speed of sound through the liquid - the greater the speed of sound, the lower the travel time. The speed of sound in any liquid is determined by the density, compressibility of the liquid, and the temperature.

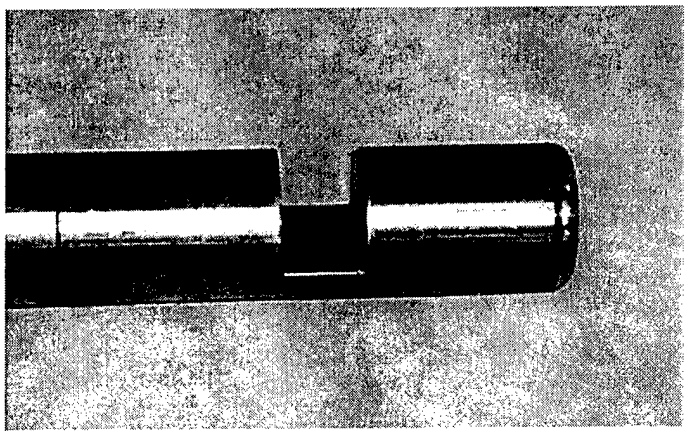

Figure 1
Ultrasonic probe gap

The TSA will operate on any process liquid in the kettle that will carry the sonic waves between the transmitting and receiving sensors without severe attenuation. Almost all liquids will carry a sonic wave. Problems will occur only when the liquid has an excess of small gas bubbles, which scatter the sonic beam, lowering the received amplitude (see note in calibration section).

## INSTRUMENT DESIGN AND INSTALLATION

### Probe and Monitor Details

Figure 2 shows the design of the TNT Slurry Analyzer probe installed at IAAP. All exterior surfaces that contact the TNT melt are made of stainless steel. The 1 MHz ultrasonic sensing transducers (item 16) are contained within the lower section of the probe, behind a 0.188-in. wall of stainless steel. These walls are separated by 1 in. to form a gap that the ultrasonic waves pass through. This lower section is laser welded to the upper section so that the entire assembly is sealed. Signal cables (item 12) pass through the center of the probe and exit through the electrical conduit fitting at the top.

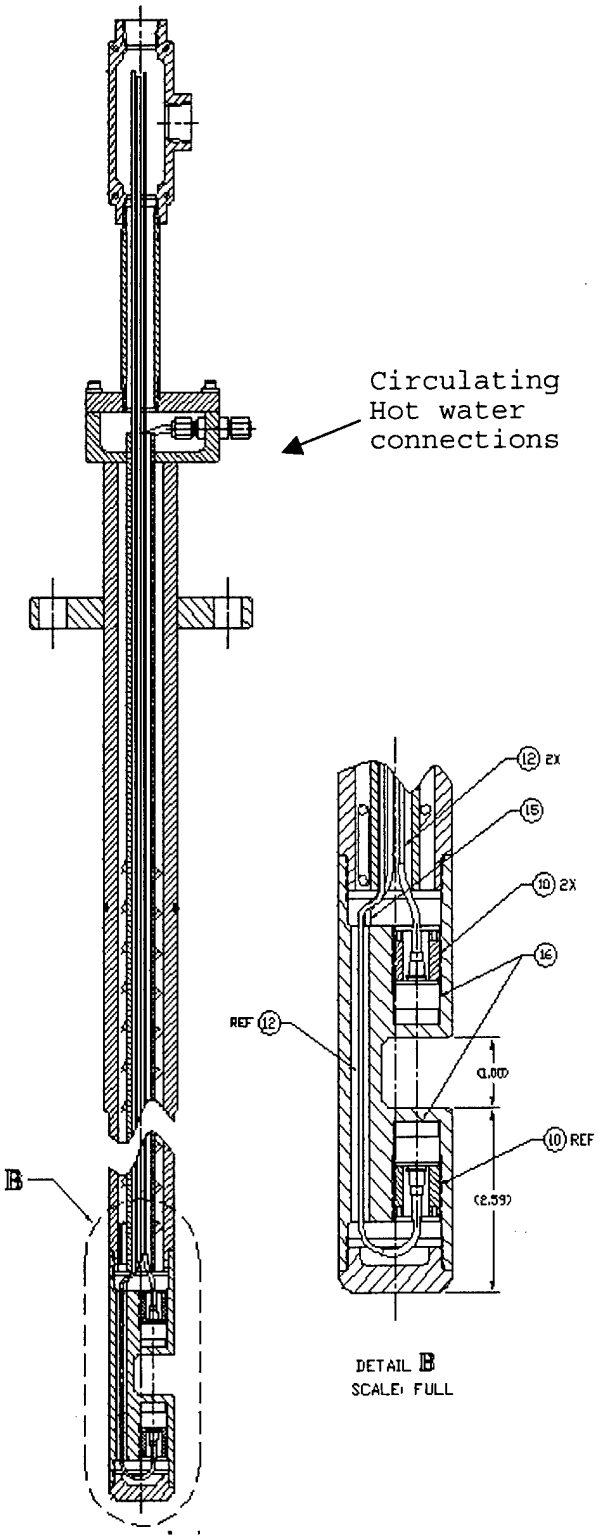


Figure 2
Probe for the TNT slurry analyzer at IAAP

A heating coil is located within the probe, and is sealed from the sensor section at the bottom. Hot water can be circulated though this heating coil to prevent TNT solids from forming on the outer walls of the probe. The ultrasonic waves pass though the mixture filling the gap at the lower portion of the probe. The waves are transmitted by one sensor and then received by another sensor after passing though the TNT mixture. Signals from the receiving sensor are then analyzed to determine the solids concentration.

The TSA probe is equipped with a three-wire resistance temperature detector (RTD) measurement interface. This RTD sensor is located in the support section near the gap (at location "ref 12" in figure 2). The TOF readings taken by the TSA are sensitive to the temperature of the process liquid, so temperature would normally be measured and then adjusted for in the solids calculation. However, in the case of TNT solids, the temperature of the melt is fixed at the TNT melt temperature during the addition of solids flake (82°C, 180°F). Since there is no temperature change when melting solid flakes are present, there is no need to adjust the sonic TOF readings for temperature changes. The TOF readings are **a direct measure of TNT solids level.**

Figure 3 shows the positioning of the probe in the 50-gal kettle used to conduct the initial testing at ARDEC. The probe was inserted into the kettle through an existing port with a 6-in. diameter flange. To add additional rigidity, the top section of the probe has a greater wall thickness than in the gap section. The length and diameter of the probe were designed to ensure a minimum of 1.93 in. clearance for the moving kettle blades.

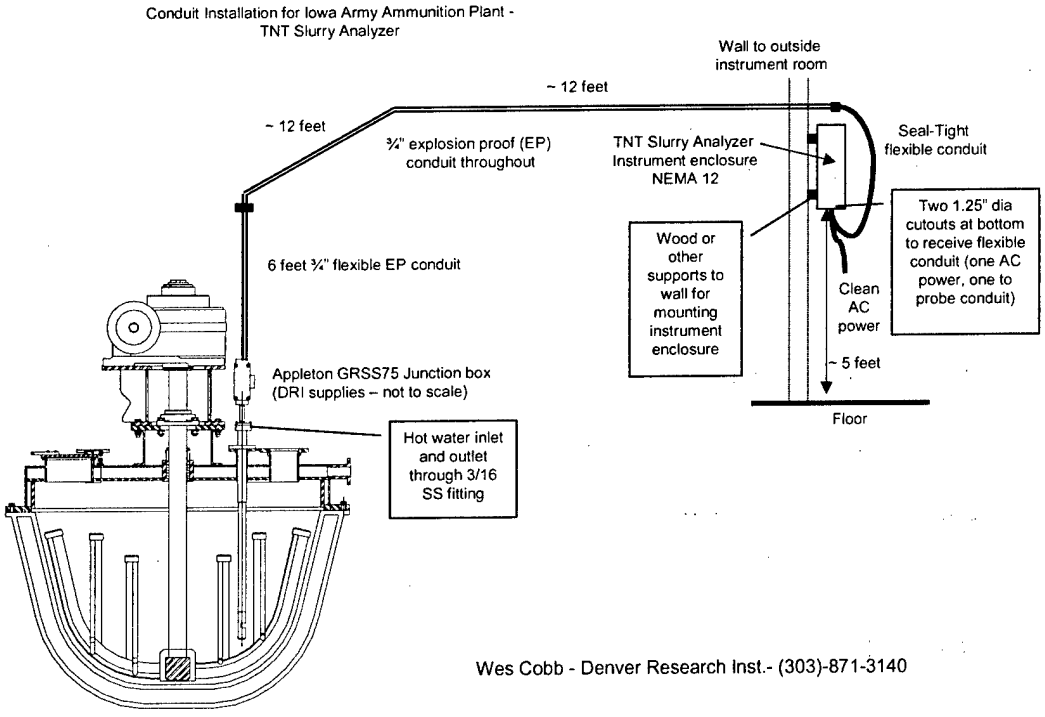


Figure 3
Placement of the probe and monitor enclosure for the south production kettle at IAAP

**Monitor Unit**

The TSA system includes:

- A stainless steel probe that contains the ultrasonic sensors and the temperature detector (described in previously)
- One monitor unit with an operator interface panel
- Sensor cables for connection to the monitor unit

The monitor unit is the heart of the TSA. The electronic boards and process computer in the monitor unit acquire the sonic and temperature readings, process this data, and provide a calibrated output for the mix composition. As shown in figure 4, the monitor unit is housed in a NEMA 4X enclosure. All connections are made through the bottom to terminal strips inside. The operator interface is mounted on the lid.

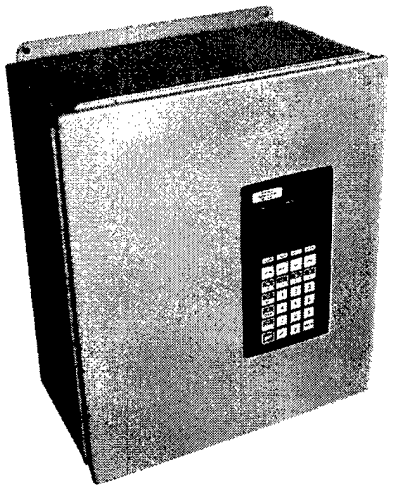

Figure 4
TSA monitor unit

The TSA is equipped with an operator interface mounted on the front panel. It is used for both installation and operation. Menu driven displays guide the operator and permit entry of calibration data. In monitor mode, the terminal displays the measured data, including solids concentration, sonic TOF readings, and temperature.

Standard outputs for the TSA include two 4 to 20 mA current loops, one for monitoring mixture concentration and one for signal amplitude. In addition, the unit provides two alarms for over-range conditions.

## Sensor Cables

Sensor cables connect the sensors to the monitor unit, which may be located some distance away (up to 40 ft is standard). Because of the high-frequency signals carried by these cables, they should not be placed near any equipment, which may generate high-frequency noise; e.g., communications or pump motors. If possible, the sensor cables should be installed in electrical conduit back to the monitor unit. If these conduits carry only the sensor and RTD signals, the shielding of the conduit will help assure a noise-free signal, and permits a greater separation of the sensors from the monitor unit.

## Safety Review and Maintenance Issues

Safety Management Systems, West Jordan, Utah, carried out a process hazards analysis of the TNT Slurry Analyzer. This analysis resulted in several safety recommendations, which were incorporated into the analyzer design. These are detailed in the hazards report (ref. 3).

## Calibration

The TSA was calibrated in the 50-gal pilot scale melt kettle at ARDEC by adding increasing amounts of TNT flake to the pilot kettle while recording the analyzer TOF and amplitude readings. Multiple analyzer readings were taken at a constant temperature, right at the melt point, as the solids concentration was raised. This data was used to establish a TOF versus solids-concentration relationship. This curve can be expressed as:

$$TOF\ (\mu s) = A_S + B_S{*}Solids(\%) \qquad (1)$$

Figure 5 shows the initial calibration obtained at ARDEC in July 2001. The TOF is a linear function of the solids level and decreases as the solids increase. The calibration coefficients, $A_S + B_S$, are obtained from a linear regression of the data points (ref.1). For the initial calibration shown in figure 5, a maximum of 15% solids by weight could be added to the heel. Above 15%, the slurry becomes very thick and air bubbles are pulled into the mix, causing severe attenuation of the ultrasound signal. This means that the signal level was too low to obtain an accurate analyzer reading. More recently, ARDEC has added chemical surfactants (Span 85) and measured slurries with added solids up to 23% by weight.

Once calibrated, the analyzer uses equation 1 to display the solids concentration for each measured TOF. The calibration coefficients in equation 1 can be used for any TNT analyzer, as long as the dimensions of the liquid gap and probe wall thickness are the same. Since the temperature of the TNT mix is fixed at 180°F during solids addition, temperature does not affect the TOF reading. Only the percent of solids changes the TOF.

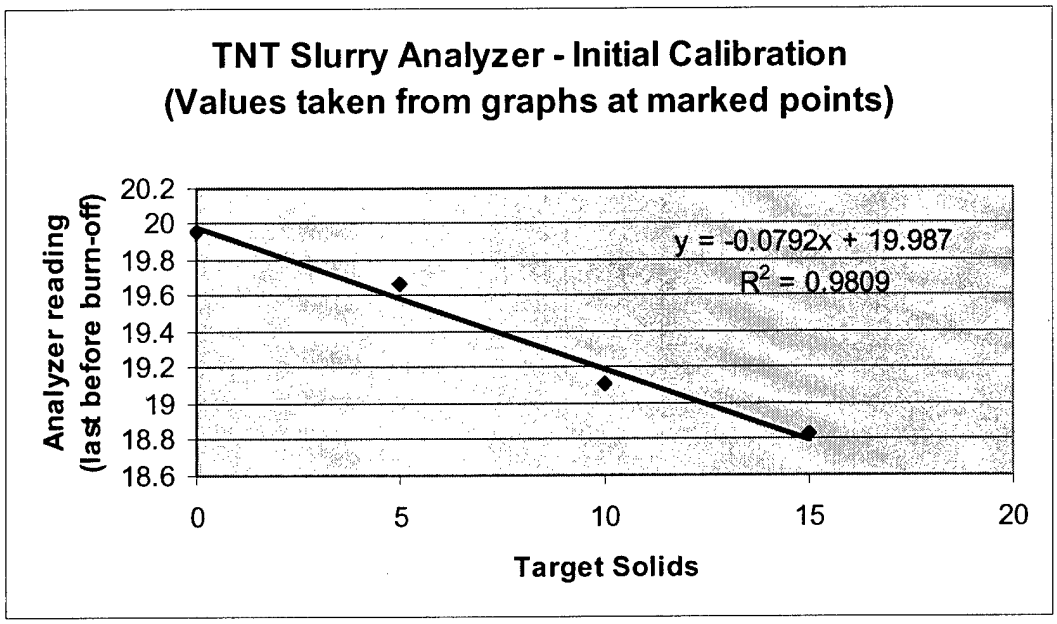


Figure 5
Initial calibration of the TNT slurry analyzer at ARDEC

It is important to note that any calibration of the TSA holds only for the material used in that calibration. Thus, if the chemical properties of the TNT used for one batch differ significantly from those of a second batch, a calibration developed for the first cannot be expected to give the correct calculated solids using the second TNT type. However, for the TNT batches used to date, no significant differences in material properties have been noted. Multiple runs, **at ARDEC**, using different TNT lots have resulted in similar calibration factors.

## TESTING

The TSA was first installed in the "south" kettle on Line 3A of IAAP during the week of 8 April 2002. However, a small leak in the connections for the circulating hot water was discovered during testing, and it could not be repaired on site. The leak was repaired and the unit was returned to IAAP for installation on 17 May 17 2002.

Installation went smoothly and the probe was inserted into the kettle through the four-bolt flange in the lid; the hot water for the internal heating jacket was connected to the new 1/8-in. female pipe connections on the probe and the electrical connections were installed back to the equipment room. The probe operated as designed, and no safety issues arose. To confirm proper operation of the TSA, the kettle was filled with hot water and the TOF and signal amplitude were recorded with water filling the probe gap. For a water temperature of 58.2°C, the TOF measured 19.299 μs, and the signal amplitude was 1.88 V. These tests can be repeated whenever the proper operation of the TSA needs to be confirmed.

The first testing phase collected readings from the TSA for the standard mix parameters used for projectile loading. Continuous readings were recorded manually from the analyzer display starting with the TNT liquid heel, throughout the addition of TNT solids feather, and stopping only after all solids had melted out.

The goal of the second test phase was to establish initial process control limits based on the input (TSA instrument readings) versus the outputs (radiographic results and pour times). American Ordnance (AO) has successfully completed most of the phase 2 testing and some preliminary results are included in this report (ref. 4). For this phase, the TSA readings were output electronically to the AO process control computer so that the readings could be recorded automatically rather than manually.

The third testing phase, which was developed by AO, will confirm that the process control limits established in phase 2 results in good quality projectiles. A number of production pours will be used for this phase.

## RESULTS

### Phase I

The phase I TOF readings are shown in figure 6. The first readings were for the liquid TNT heel that was just covering the probe gap. TNT solids flake was then feathered into the kettle from the shaken hopper above the kettle. In figure 6, the solids increase is shown as the **decrease** in measured TOF because the TOF is inversely related to solids content. However, there was a sharp decrease in the amplitude of the ultrasonic signal going thought the probe gap after about half of the solids flake was mixed in.

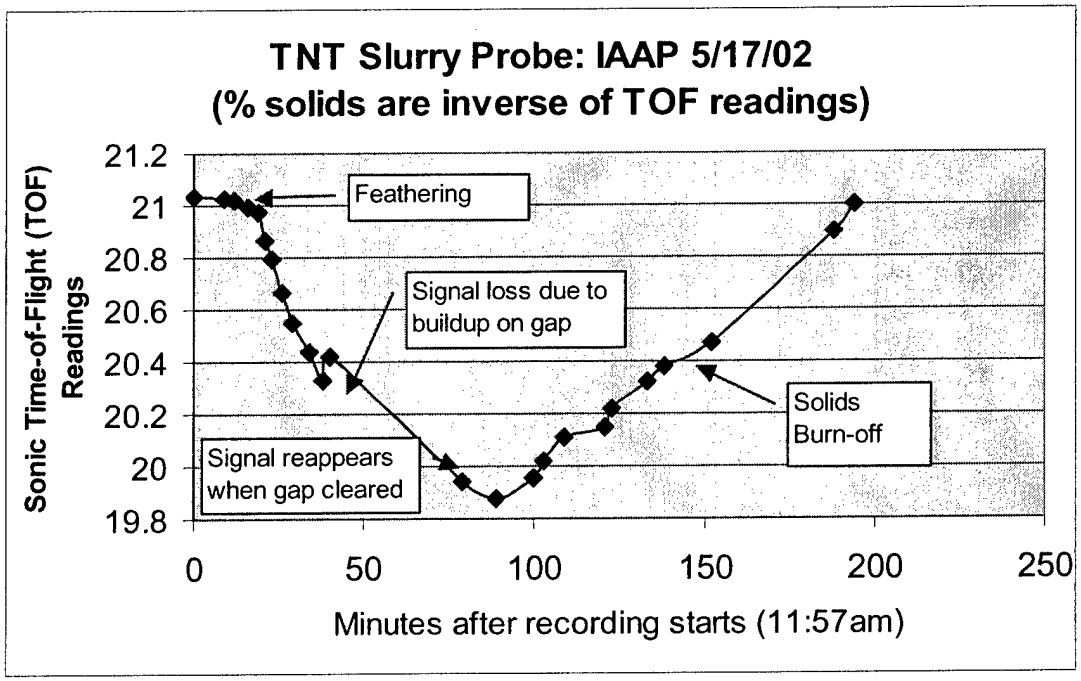


Figure 6
Initial TOF readings for the TSA just after installation

The signal amplitude recovered just after all solids were in and the blade speed was increased. It is suspected that the probe gap was blocked, but it could not be confirm because the probe was submerged. Increasing the blade speed probably knocked out the blockage. Note that the blockage of the probe was seen at ARDEC when the heating coil was not yet connected. Once connected and the probe surface was adequately heated, blockage was not a problem.

Just after all solids were in, a strong signal amplitude (1.68 V) and a good ultrasonic reading was recorded. At this point, the slurry was judged to be too thick to pour. Normally, solids burn-off would continue until the operator judged the slurry to be at the proper consistency. These tests confirmed that the analyzer could read the solids for the typical slurry thickness at IAAP. The blockage only prevented readings during solids feathering, not afterwards when the solids must be measured.

After the slurry reached full thickness, measurements were continued as solids were slowly burnt off. The kettle temperature was the same as that during feathering. The analyzer showed a steady decrease in solids during this 2-hr period, confirming our expectations. Once all solids were burnt off, the kettle was poured and the condition of the probe could be seen. Even after slow burn out, there was still a 2-in. buildup of solids stuck to the leading edge of the gap. This buildup extended part way into the gap and would certainly reduce signal amplitude. For example, note that the final amplitude after burn off (1.43) is lower than that for heel alone (2.04). A long buildup of solids on the leading edge above the lower 6-in. (gap) portion of the probe (fig. 7) was also noted. This buildup extended for about 2 ft along the probe.

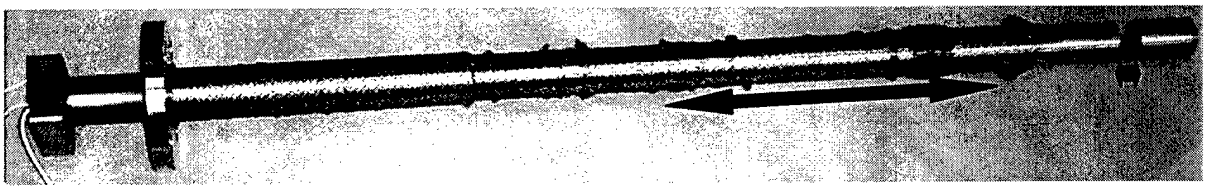

Figure 7
TSA probe showing areas of buildup/blockage

As noted previously, entrapped air can significantly reduce the signal amplitude and prevent good TSA readings. During the initial tests at the pilot kettle at ARDEC, lots of air was trapped in the slurry when the solids content was raised above the normal amount. However, during the initial IAAP tests, there was no evidence of air being trapped in the slurry during either the addition of solids or burn off. Trapped air would have been seen as bubbles coming out of the mix during the slow burn off.

Using the calibrations established during the pilot tests at ARDEC, the TOF readings can be converted to a percent solids measurement. If the TOF versus solids calibration factor of –0.0792 (fig. 5) is used and solids are assumed to be zero after burn-off, the solids readings in figure 8 can be calculated from equation 1 (no temperature component).

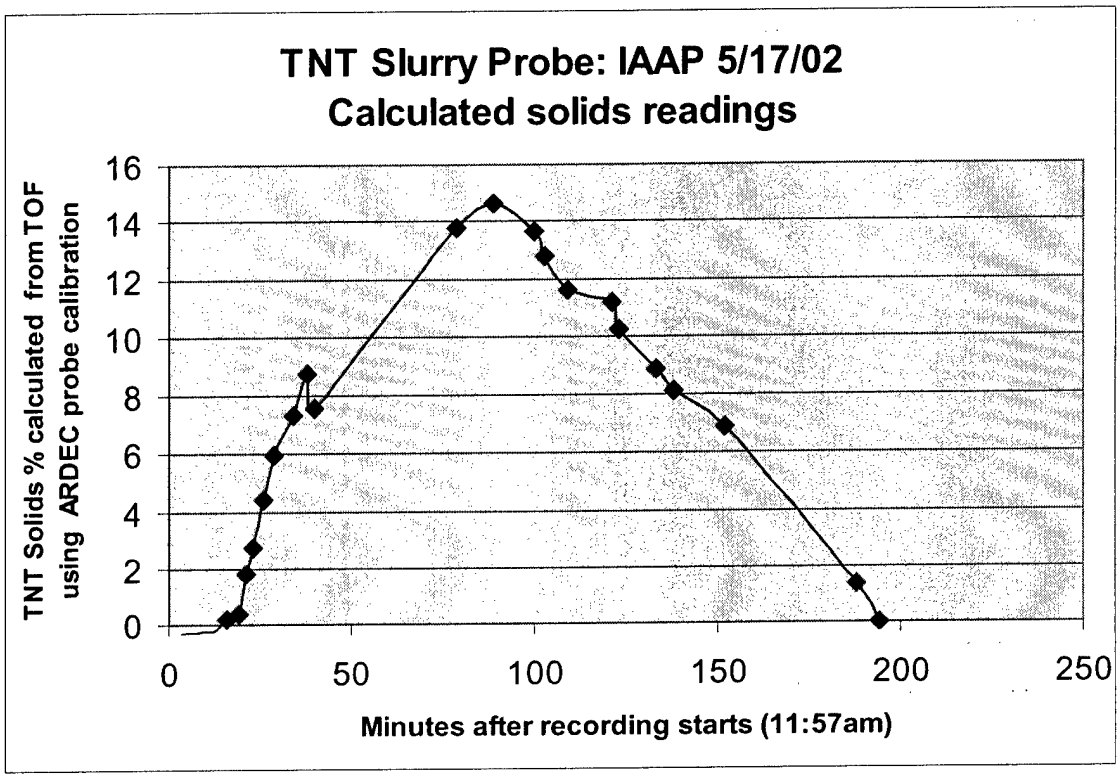


Figure 8
TNT solids calculated from the TOF readings in figure 6

Although the calculated solids show the expected increase during TNT solids feathering and decrease during burn-off, the maximum calculated solids is only about 15%. This is significantly below the expected range for solids percent, which would be 25 to 30% based on the amount added and assuming no burn-off during feathering. The lower than expected calculated solids may be caused by the following:

- The calibration factor established at ARDEC may be inaccurate because the actual solids at the time of TOF reading may have been **higher** than that based on the amount of solid flake added. For these calibration runs, the pilot kettle was purposely kept cooler than normal and an actual increase in solids was often observed after the flake was added.

- The preliminary calibration developed at ARDEC may not apply for the TNT properties used at IAAP.

- The assumption that all solids had been burnt off for the last reading may not be true. Thus, the last point, which set the 0% solids point, may actually have been a few percent higher. This would shift the curve upwards by that extra percent. Although no flake was seen in the melt, the data does not tail off to a vertical line as it should for no solids.

Unfortunately, the data recording stopped prematurely. Future test runs that record the TOF during extended burn-off could answer this question.

Although the accuracy of the preliminary calibration may be in question, this does not impact the intended use of the TSA. American Ordnance plans to **use the TSA readings directly** to control process operations, rather than a calculated solids reading. Through the phase II and phase III testing discussed previously, they will establish control limits for the kettle pour based on the TOF readings from the TSA. As long as the pour is made while the TOF is within these limits, the quality of the munition is assured.

**Phase II**

The purpose of the phase II tests is to establish process control limits based on TSA instrument readings. Details of these tests can be found in the AO report and are not duplicated here. These tests varied the pour time to cast munitions with different TNT solids loadings (recall that solids burn off over time in the kettle). Pour time is the time from the start of the solids feather addition to the kettle opening. If the solids are too high or low, the munitions are expected to have poor cast quality; i.e., porosity or cracks. The TSA readings were monitored and later compared with the results of radiographic inspection. This comparison established initial process control limits based on the TSA readings.

Figure 9 shows TSA readings for the first phase II run. Note that several probe-heating improvements were made for phase II to prevent buildup on the sensor gap. Improved heating may be the reason for reduced signal loss during the feathering.

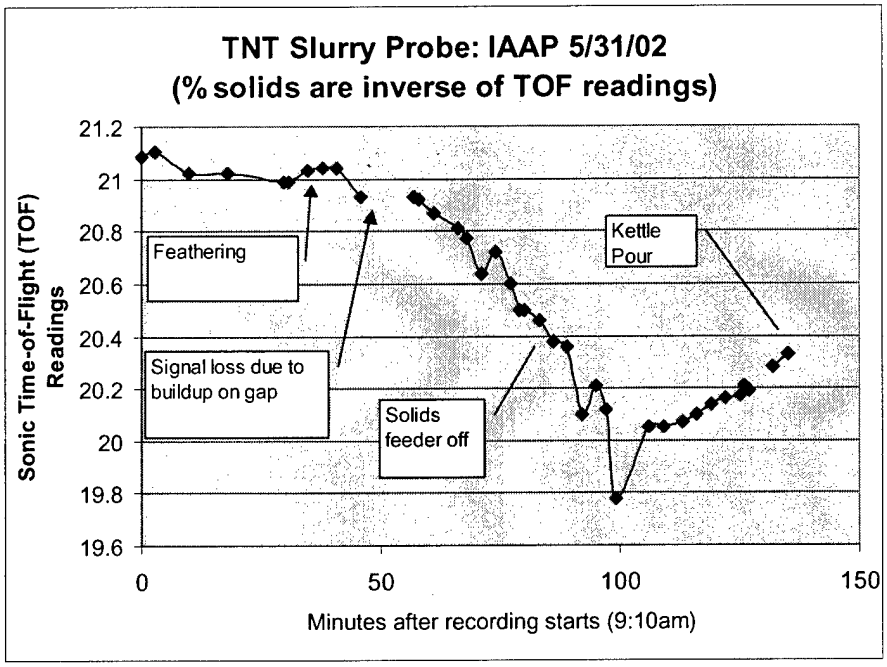


Figure 9
TSA TOF readings for second phase tests

A second kettle pour was run to determine the optimum pouring time based on the TSA readings. The TOF results showed a similar decrease as solids feathered in. Once all the solids were in, three pours were made at successively later times. Later, all radiographic inspection of the shells from the three pour times was acceptable.

## CONCLUSIONS

The TNT Slurry Analyzer (TSA) is working at Iowa Army Ammunition Plant and is measuring solids content in the mix in real time. This information is used to monitor the quality of the mix and determine when optimal pour conditions are reached.

Using data from the phase II runs, American Ordnance (AO) has established initial, upper and lower control limits for the TSA time-of-flight readings that result in good munitions casts (TOF of 19.9 and 20.2, respectively). In phase III, AO will continue to confirm additional munitions castings based on the TSA readings.

## RECOMMENDATIONS

1. Increase the "averaging setting" of the TNT Slurry Analyzer (TSA) to 1 min and read data once per minute. The TSA is currently set to 10 sec averaging and a data point is read every 3 min. Increasing the averaging will reduce the fluctuations in measurements that are caused by real variations in the solids passing through the gap.

2. Perform periodic inspection of the probe assembly. As stated in the recommendations of the Hazards Analysis, the welds on the probe should be routinely checked using dye penetrant testing. Denver Research Institute recommends that these welds be checked at 6-month intervals.

3. Test the TSA monthly with water in the kettle to make sure there are no changes in signal amplitude response.

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4. Benson, T. and Fry, B., "Slurry Analyzer Tests Phase II," American Ordnance, draft dated 24 Jun 2002.

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